

Kentucky Method 64-257-02
Revised 12/23/02
Supersedes 64-257-2000
Dated 1/13/00

ANALYSIS FOR HINDERED AMINE LIGHT STABILIZER (HALS)

1. SCOPE: This method is designed to determine the presence of hindered amine light stabilizer (HALS) in the polyol portion of a two component acrylic urethane coating.
2. APPARATUS AND MATERIALS:
 - 2.1. Gas Chromatograph with Mass Spectrometer Detector
 - 2.2. 5% Phenyl Methyl Siloxane Capillary Column
 - 2.3. Reference HALS sample
 - 2.3. Hexane, HPLC grade
 - 2.5. Centrifuge
3. INSTRUMENT CONDITIONS: Install and condition the column in the chromatograph in accordance with manufacturers recommendations. Establish operating conditions yielding acceptable separation of the components, seeing Table 1 as a guide for initial instrument conditions. Values may need to be altered to achieve necessary resolution of components. Allow sufficient time for the instrument to reach equilibrium and to produce a stable base line.

TABLE 1

INJECTION PORT	
Mode	Splitless
Carrier gas	Helium
Initial temperature	250 °C
Pressure	7.6 PSI
Total flow	54 ml/min
Purge flow	50 ml/min
Purge time	2 min
Injection volume	1.0 µl
COLUMN PARAMETERS	
Nominal length	30 m
Nominal diameter	0.25 mm
Nominal film thickness	0.25 µm
Maximum temperature	325 °C
Mode	Constant flow
Initial flow	1.0 ml/min
OVEN PARAMETERS	
Initial temperature	50 °C
Maximum temperature	325 °C
Equilibration time	0.5 min
Initial time	1.0 min
Temperature Ramp 1	25.0 °C/min to 250 °C
Hold time 1	15.0 min
Temperature ramp 2	50.0 °C/min to 300 °C
Hold time 2	5.0 min
MASS SPECTROMETER	
Transfer line heater	280 °C
Solvent delay	2.0 min

4. PROCEDURE:

- 4.1. Prepare standards by adding reference HALS compounds to Hexane to obtain the desired concentrations. Record the weights of Hexane and reference HALS used to the nearest 0.1 mg. These standards will be used to obtain retention time data and atomic masses incidental to the specific HALS.
- 4.2. Prepare samples by thoroughly mixing 2 parts Hexane to 1 part polyol in a centrifuge tube. 10.0 ml of Hexane and 5.0 ml of polyol is a suitable working volume. Record the weights of Hexane and sample polyol used to the nearest 0.1 mg. Cap the centrifuge tube and centrifuge for 5 minutes at a minimum relative centrifugal force (RCF) of 43. See 5.1 for RCF equation. Fill a sample vial with the resulting supernate.

- 4.3. Inject a 1.0 µl aliquot of the appropriate prepared reference HALS standard. Determine the retention times for the associated peaks and the atomic masses of the reference HALS compounds.
- 4.4. Inject a 1.0 µl aliquot of the prepared sample. Determine qualitative presence of the specific HALS by comparing retention times and verifying atomic masses of the HALS compounds.
- 4.5. Quantitative determinations may be made with the addition of an appropriate internal standard.

5. CALCULATION FOR RELATIVE CENTRIFUGAL FORCE (RCF):

$$RCF = 0.00001118 \times r \times N^2$$

Where, RCF = Relative Centrifugal Force
r = Rotating radius (cm)
N = Revolutions per minute

6. REPORT:

- 6.1. Report qualitative results as Pass or Fail.
- 6.2. Report quantitative results to the nearest 0.01 %.

APPROVED _____
Director
DIVISION OF MATERIALS

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